Bis(guanidinium) 5-sulfosalicylate monohydrate

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The crystal structure of the proton-transfer compound hydrate of 3-carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid) with guanidine, $[2(C\ H_6N_3)^+\ (C_7H_4O_6S)^{2-}.\ (H_2O)]$ (I), reveals the presence of an extensively hydrogen-bonded three- dimensional network polymer in which all proton donor and acceptor atoms on the two guanidine cations, the dianionic sulfosalicylate anion and the lattice water molecule are utilized. The structure is significanty different chemically and structurally from that of the previously reported anhydrous guanidinium sulfosalicylate compound $[(C\ H_6N_3)^+\ (C_7H_5O_6S)^-]$.

Experimental

Crystal data

$C_7H_4O_6S·2(CH_6N_3)·H_2O$	V = 1579.5 (7) Å^3
M_r = 354.35	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71069 Å
a = 12.030 (3) Å	μ = 0.25 mm^{-1}
b = 18.010 (5) Å	T = 295 K
c = 7.290 (2) Å	0.45 × 0.35 × 0.30 mm

Data collection

Rigaku AFC7R diffractometer	1644 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan *TEXSAN* for Windows (Molecular Structure Corporation, 1999)	R_{int} = 0.028
T_{min} = 0.895, T_{max} = 0.928	3 standard reflections every 150 reflections
2428 measured reflections	intensity decay: 0.3%
2173 independent reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.140	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
S = 0.90	$\Delta\rho_{min}$ = −0.45 e Å^{-3}
2173 reflections	Absolute structure: Flack (1983) 79
261 parameters	Flack parameter: 0.1 (2)
0 restraints	

Table 1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.98 (6)	1.62 (6)	2.518 (3)	152 (5)
O1*W*—H1*A*···O72^{i}	0.90 (10)	2.11 (10)	3.008 (7)	179 (9)
O1*W*—H1*B*···O53^{ii}	0.93 (11)	2.13 (11)	3.055 (7)	180 (8)
N11—H11*A*···O53	0.81 (7)	2.29 (7)	3.018 (7)	151 (5)
N11—H11*A*···O2^{iii}	0.81 (7)	2.71 (7)	3.079 (7)	110 (6)
N11—H11*B*···O51^{iv}	0.90 (5)	2.19 (5)	3.089 (7)	179 (6)
N12—H12*A*···O72^{iii}	0.90 (4)	2.09 (4)	2.987 (7)	179 (6)
N12—H12*B*···O52^{v}	0.89 (5)	2.17 (5)	3.006 (6)	157 (4)
N21—H21*A*···O52^{vi}	0.83 (5)	2.52 (5)	3.127 (6)	131 (5)
N21—H21*A*···O2^{iii}	0.83 (5)	2.34 (5)	3.031 (6)	141 (5)
N21—H21*B*···O51^{vii}	0.89 (6)	2.21 (6)	3.003 (7)	149 (5)
N22—H22*A*···O71^{i}	0.90 (7)	1.94 (7)	2.801 (6)	159 (6)
N22—H22*B*···O52^{v}	1.00 (9)	2.22 (9)	3.043 (7)	139 (7)
N32—H32*A*···O1*W*	0.94 (9)	1.93 (9)	2.871 (8)	180 (8)
N32—H32*B*···O71^{iii}	0.81 (7)	2.06 (8)	2.826 (8)	157 (8)
C4—H4···O51	0.95	2.59	2.953 (5)	103

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*+1/2, −*y*, *z*+1/2; (iv) *x*−1/2, −*y*+1/2, −*z*+1; (v) *x*+1/2, −*y*+1/2, −*z*+1; (vi) *x*, *y*, *z*+1; (vii) *x*−1/2, −*y*+1/2, −*z*+2.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999); data reduction: *TEXSAN* for Windows (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* for Windows (Spek, 1999); software used to prepare material for publication: *PLATON* for Windows (Spek, 1999).

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supplementary materials

Bis(guanidinium) 5-sulfosalicylate monohydrate

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Comment

The acid strength of aromatic sulfonic acids ensures that the formation of proton-transfer compounds will result from their reaction with most Lewis bases. Furthermore, the sulfonate group provides three available oxygen atoms as proton-accepting centres for hydrogen-bonding associations, enhancing the utility of sulfonates for molecular assembly. The structures of the guanidinium salts of a number of sulfonates including aromatic types have been determined because of their potential as optical materials (Russell *et al.*, 1994a; Russell *et al.*, 1994b). This property arises through a combination of the trigonal symmetry of the guanidinium cation with that of the sulfonate group, giving assembly into 2-dimensional hydrogen-bonded sheet structures which may be extended through inter-layer linkages via the third sulfonate-O into 3-imensional network polymers.

3-Carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid = 5-SSA) has the additional interactive carboxylic acid and phenolic functional substituent groups which lend themselves to secondary n-dimensional hydrogen-bonding extension. Furthermore, 5-SSA is able to protonate water and several hydrate structures of the acid have been reported: the dihydrate (Attig & Mootz, 1977; Aliev *et al.*, 1995), the dideuterate (Attig & Williams, 1977), the trihydrate (Attig & Mootz, 1977), and the pentahydrate (Merschenz-Quack & Mootz, 1990). Many of these have protonated polyaqua species, such as the $H_7O_3^+$ cation species of the trihydrate (Mootz & Fayoz, 1970). The only reported structures of Lewis base 5-SSA proton-transfer compounds are with aniline (an anhydrate) (Bakasova *et al.*, 1991), theophylline (a monohydrate) (Madarasz *et al.*, 2002), trimethoprim (a dihydrate) (Raj *et al.*, 2003), and guanidine (GU), a 1:1 anhydrate (Zhang *et al.*, 2004). In addition, we have also determined the structures of the proton-transfer compounds of 5-SSA with a set of bicyclic heteroaromatic Lewis bases (Smith *et al.*, 2004). These are with quinoline: quinolinium 5-sulfosalicylate trihydrate; 8-hydroxyquinoline: 8-hydroxyquinolinium 5-sulfosalicylate monohydrate; 8-aminoquinoline: 8-aminoquinolinium 5-sulfosalicylate dihydrate; and quinaldic acid, the adduct structure quinolinium-2-carboxylic acid 5-sulfosalicylate-quinoline-2-carboxylic acid (1/1). We also prepared a compound with guanidine which is a (2:1) hydrate, reported here: $[2(GU)^+ (5\text{-}SSA)^{2-}. H_2O]$ (I), which is significantly different both chemically and structurally from the Zhang *et al.* (2004) structure.

The structure of (I) shows the presence of a dianionic ($5\text{-}SSA)^{2-}$ species, arising from the deprotonation of both the sulfonate and the carboxylic acid groups (Fig. 1). As expected with guanidinium salts, there is extensive hydrogen bonding, in this case involving all potential H-donors of the GU^+ cation in 14 interactions to all sulfonate-O, both carboxylate-O, the phenolic-O and the water-O acceptor sites of the $(5\text{-}SSA)^-$ anions (Table 1). However, no cyclic $R^2_2(8)$ N—H (guanidine)···O (sulfonate) interactions are present such as are found in the previously reported $[(GU)^+ (5\text{-}SSA)^-]$ structure (Zhang *et al.*, 2004), where a short O—H···O(carboxyl) linkage [2.611 (2) Å] is also found. The result in (I) is a three- dimensional network polymer structure (Fig. 2).

The usual intramolecular O (phenol)···O (carboxyl) hydrogen bond is present [O···O, 2.518 (3) Å] in the 5-SSA anion molecule, this distance comparing with 2.601 (2) Å in the 1:1 compound (Zhang *et al.*, 2004).

Experimental

The synthesis of the title compound were carried out by heating under reflux, 1 mmol quantities of 3-carboxy-4-hydroxybenzenesulfonic acid [5-sulfosalicylic acid (5-SSA)] and guanidine carbonate in 50 mL of 50% ethanol/water for 10 min. After concentration to ca. 30 mL, partial room temperature evaporation of the hot-filtered solution gave large colourless flat prisms of (I) (m. pt. range, 478.5–484.1 K).

Refinement

All hydrogen atoms involved in hydrogen-bonding interactions (those on the guanidinium cation and on the lattice water) were located by difference methods and their positional and isotropic thermal parameters were refined. Others were included in the respective refinements at calculated positions (C—H = 1.95 Å) as riding models, with U_{eq} fixed at $1.2U_{eq}$ (atom).

(683GS03_SSAGU)

Crystal data

$C_7H_4O_6S·2(CH_6N_3)·H_2O$	D_x = 1.490 Mg m^{-3}
M_r = 354.35	Melting point: 478.5–484.1 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71069 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 12.030 (3) Å	θ = 12.5–17.4°
b = 18.010 (5) Å	μ = 0.25 mm^{-1}
c = 7.290 (2) Å	T = 295 K
V = 1579.5 (7) Å^3	Prism, Colourless
Z = 4	0.45 × 0.35 × 0.30 mm
F(000) = 744	

Data collection

Rigaku AFC7R diffractometer	1644 reflections with $I > 2\sigma(I)$
Radiation source: Rigaku rotating anode	R_{int} = 0.028
graphite	θ_{max} = 27.5°, θ_{min} = 2.8°
ω–2θ scans	h = −6→15
Absorption correction: ψ scan *TEXSAN* for Windows (Molecular Structure Corporation, 1999)	k = 0→23
T_{min} = 0.895, T_{max} = 0.928	l = −3→9
2428 measured reflections	3 standard reflections every 150 reflections
2173 independent reflections	intensity decay: 0.3%

Refinement

Refinement on F^2	Hydrogen site location: Inferred from neighbouring sites
Least-squares matrix: Full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 2.556P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.140$	$(\Delta/\sigma)_{max} = 0.015$
$S = 0.90$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
2173 reflections	$\Delta\rho_{min} = -0.45$ e Å^{-3}
261 parameters	Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.059 (4)
Primary atom site location: Structure-invariant direct methods	Absolute structure: Flack (1983) 79
Secondary atom site location: Difference Fourier map	Flack parameter: 0.1 (2)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S5	0.31812 (7)	0.21494 (5)	0.5031 (2)	0.0359 (3)
O2	0.2675 (2)	−0.11002 (13)	0.5046 (7)	0.0456 (9)
O51	0.4352 (2)	0.23381 (15)	0.5074 (7)	0.0484 (9)
O52	0.2646 (3)	0.2396 (2)	0.3331 (5)	0.0487 (12)
O53	0.2590 (3)	0.2426 (2)	0.6645 (5)	0.0467 (12)
O71	−0.0090 (2)	0.02380 (17)	0.5051 (8)	0.0641 (12)
O72	0.0603 (2)	−0.09012 (15)	0.5092 (8)	0.0548 (9)
C1	0.1857 (3)	0.01113 (18)	0.5073 (8)	0.0320 (9)
C2	0.2797 (3)	−0.03504 (18)	0.5055 (9)	0.0343 (9)
C3	0.3852 (3)	−0.0046 (2)	0.5030 (11)	0.0426 (10)
C4	0.3993 (3)	0.0716 (2)	0.5026 (9)	0.0390 (10)
C5	0.3066 (3)	0.11746 (18)	0.5068 (7)	0.0326 (9)
C6	0.2013 (3)	0.08740 (18)	0.5108 (8)	0.0325 (9)
C7	0.0706 (3)	−0.0196 (2)	0.5083 (11)	0.0432 (11)
N12	0.6373 (4)	0.1236 (3)	0.7752 (8)	0.0573 (19)
N22	0.8109 (4)	0.0943 (3)	0.6753 (8)	0.0620 (16)
N32	0.6961 (5)	0.0027 (3)	0.7800 (10)	0.080 (2)
C12	0.7150 (4)	0.0736 (3)	0.7432 (7)	0.0507 (14)
N11	0.0412 (5)	0.2039 (3)	0.8465 (8)	0.0513 (17)
N21	0.0364 (4)	0.1951 (3)	1.1595 (8)	0.0550 (19)

N31	−0.1173 (3)	0.23667 (19)	1.0044 (8)	0.0455 (10)
C11	−0.0130 (3)	0.21230 (19)	1.0045 (9)	0.0378 (10)
O1W	0.8614 (5)	−0.1118 (3)	0.7534 (9)	0.0727 (18)
H2	0.188 (5)	−0.117 (3)	0.526 (9)	0.066 (16)*
H3	0.448400	−0.036300	0.501600	0.0510*
H4	0.471700	0.092400	0.498000	0.0470*
H6	0.138300	0.119300	0.519000	0.0390*
H12A	0.578 (3)	0.114 (3)	0.846 (6)	0.065 (10)*
H12B	0.655 (4)	0.169 (3)	0.739 (7)	0.061 (14)*
H22A	0.864 (6)	0.062 (4)	0.639 (9)	0.09 (2)*
H22B	0.837 (7)	0.147 (5)	0.677 (12)	0.10 (3)*
H32A	0.750 (7)	−0.035 (5)	0.771 (11)	0.11 (3)*
H32B	0.635 (6)	−0.009 (4)	0.817 (12)	0.09 (2)*
H11A	0.106 (6)	0.215 (4)	0.838 (9)	0.08 (2)*
H11B	0.010 (4)	0.222 (3)	0.744 (7)	0.061 (15)*
H21A	0.103 (4)	0.184 (3)	1.160 (7)	0.065 (14)*
H21B	0.006 (5)	0.198 (3)	1.270 (8)	0.060 (16)*
H1A	0.921 (9)	−0.105 (6)	0.681 (10)	0.10 (3)*
H1B	0.825 (10)	−0.156 (6)	0.778 (10)	0.11 (3)*
H31A	−0.164 (5)	0.246 (5)	1.114 (9)	0.08 (3)*
H31B	−0.157 (5)	0.245 (5)	0.892 (9)	0.09 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S5	0.0386 (5)	0.0307 (4)	0.0385 (5)	−0.0045 (3)	0.0000 (8)	−0.0005 (7)
O2	0.0437 (14)	0.0272 (12)	0.0660 (18)	0.0035 (10)	0.004 (3)	0.004 (2)
O51	0.0460 (15)	0.0437 (14)	0.0554 (17)	−0.0127 (11)	−0.001 (2)	−0.002 (3)
O52	0.061 (2)	0.037 (2)	0.048 (2)	0.0053 (19)	−0.0051 (19)	0.0085 (16)
O53	0.055 (2)	0.041 (2)	0.044 (2)	−0.0067 (19)	0.0050 (18)	−0.0082 (16)
O71	0.0271 (12)	0.0512 (16)	0.114 (3)	0.0016 (11)	0.000 (3)	−0.015 (3)
O72	0.0399 (14)	0.0365 (13)	0.088 (2)	−0.0082 (11)	0.001 (3)	−0.002 (3)
C1	0.0302 (15)	0.0317 (15)	0.0340 (17)	−0.0007 (12)	0.000 (3)	−0.004 (2)
C2	0.0369 (16)	0.0302 (15)	0.0357 (18)	0.0018 (13)	0.006 (3)	0.003 (3)
C3	0.0307 (15)	0.0410 (18)	0.056 (2)	0.0075 (14)	0.002 (4)	0.003 (4)
C4	0.0281 (15)	0.0410 (18)	0.048 (2)	−0.0022 (13)	−0.003 (3)	−0.005 (4)
C5	0.0357 (16)	0.0317 (15)	0.0303 (16)	−0.0026 (12)	−0.002 (3)	0.001 (2)
C6	0.0323 (15)	0.0300 (15)	0.0352 (18)	0.0021 (12)	−0.001 (3)	−0.002 (2)
C7	0.0359 (17)	0.0407 (18)	0.053 (2)	−0.0032 (14)	0.002 (3)	−0.002 (4)
N12	0.040 (3)	0.046 (3)	0.086 (4)	0.002 (2)	0.017 (3)	0.013 (3)
N22	0.051 (2)	0.045 (2)	0.090 (4)	0.003 (2)	0.020 (3)	0.000 (2)
N32	0.059 (3)	0.047 (2)	0.134 (5)	0.007 (2)	0.021 (4)	0.027 (3)
C12	0.046 (2)	0.043 (2)	0.063 (3)	0.000 (2)	0.004 (2)	0.007 (2)
N11	0.047 (3)	0.058 (3)	0.049 (3)	0.005 (3)	0.005 (3)	−0.002 (3)
N21	0.046 (3)	0.073 (4)	0.046 (3)	0.017 (3)	−0.004 (3)	−0.009 (3)
N31	0.0389 (16)	0.0494 (17)	0.0483 (18)	0.0098 (14)	−0.003 (3)	0.002 (3)
C11	0.0374 (17)	0.0315 (16)	0.0444 (19)	−0.0001 (13)	−0.001 (3)	−0.006 (3)
O1W	0.062 (3)	0.050 (2)	0.106 (4)	0.003 (2)	0.007 (3)	0.006 (3)

Geometric parameters (Å, º)

S5—O51	1.449 (3)	N32—H32B	0.81 (7)
S5—O52	1.465 (4)	N11—C11	1.332 (8)
S5—O53	1.462 (4)	N21—C11	1.314 (8)
S5—C5	1.761 (3)	N31—C11	1.329 (5)
O2—C2	1.358 (4)	N11—H11B	0.90 (5)
O71—C7	1.236 (5)	N11—H11A	0.81 (7)
O72—C7	1.276 (5)	N21—H21A	0.83 (5)
O2—H2	0.98 (6)	N21—H21B	0.89 (6)
O1W—H1A	0.90 (10)	C1—C6	1.387 (5)
O1W—H1B	0.93 (11)	C1—C7	1.491 (5)
N12—C12	1.319 (7)	C1—C2	1.404 (5)
N22—C12	1.310 (7)	C2—C3	1.383 (5)
N32—C12	1.324 (8)	C3—C4	1.383 (5)
N12—H12B	0.89 (5)	C4—C5	1.388 (5)
N12—H12A	0.90 (4)	C5—C6	1.378 (5)
N22—H22B	1.00 (9)	C3—H3	0.9508
N22—H22A	0.90 (7)	C4—H4	0.9487
N32—H32A	0.94 (9)	C6—H6	0.9529
O51—S5—O52	112.0 (2)	C2—C1—C7	121.9 (3)
O51—S5—O53	112.0 (2)	C2—C1—C6	118.6 (3)
O51—S5—C5	108.05 (17)	O2—C2—C3	119.6 (3)
O52—S5—O53	111.3 (2)	C1—C2—C3	120.3 (3)
O52—S5—C5	106.3 (2)	O2—C2—C1	120.1 (3)
O53—S5—C5	106.8 (2)	C2—C3—C4	120.4 (3)
C2—O2—H2	103 (3)	C3—C4—C5	119.5 (3)
H1A—O1W—H1B	127 (9)	S5—C5—C4	122.0 (3)
C12—N12—H12B	114 (3)	S5—C5—C6	117.7 (3)
C12—N12—H12A	122 (3)	C4—C5—C6	120.3 (3)
H12A—N12—H12B	124 (4)	C1—C6—C5	120.9 (3)
H22A—N22—H22B	113 (7)	O71—C7—C1	119.0 (3)
C12—N22—H22B	123 (5)	O72—C7—C1	117.4 (3)
C12—N22—H22A	123 (5)	O71—C7—O72	123.7 (3)
H32A—N32—H32B	117 (8)	C2—C3—H3	119.74
C11—N31—H31A	126 (4)	C4—C3—H3	119.85
C11—N31—H31B	121 (4)	C3—C4—H4	120.30
H31A—N31—H31B	112 (5)	C5—C4—H4	120.23
C12—N32—H32B	118 (5)	C5—C6—H6	119.71
C12—N32—H32A	124 (5)	C1—C6—H6	119.39
C11—N11—H11B	118 (3)	N12—C12—N32	120.1 (5)
H11A—N11—H11B	105 (6)	N22—C12—N32	120.1 (5)
C11—N11—H11A	123 (4)	N12—C12—N22	119.8 (5)
C11—N21—H21A	120 (4)	N11—C11—N21	119.7 (4)
H21A—N21—H21B	114 (5)	N11—C11—N31	119.9 (6)
C11—N21—H21B	126 (4)	N21—C11—N31	120.4 (6)
C6—C1—C7	119.6 (3)		
O51—S5—C5—C4	−3.6 (6)	C6—C1—C2—O2	179.1 (6)
O51—S5—C5—C6	177.5 (4)	C6—C1—C2—C3	−1.5 (9)
O52—S5—C5—C4	116.7 (5)	C2—C1—C7—O72	0.6 (10)

O52—S5—C5—C6	−62.1 (5)	C7—C1—C2—O2	0.0 (9)
O53—S5—C5—C4	−124.3 (5)	C1—C2—C3—C4	0.1 (11)
O53—S5—C5—C6	56.8 (5)	O2—C2—C3—C4	179.6 (7)
C7—C1—C6—C5	−178.9 (6)	C2—C3—C4—C5	0.7 (11)
C2—C1—C7—O71	−178.2 (7)	C3—C4—C5—S5	−179.0 (5)
C6—C1—C7—O71	2.8 (10)	C3—C4—C5—C6	−0.2 (9)
C6—C1—C7—O72	−178.4 (6)	S5—C5—C6—C1	177.7 (5)
C2—C1—C6—C5	2.0 (9)	C4—C5—C6—C1	−1.2 (9)
C7—C1—C2—C3	179.4 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.98 (6)	1.62 (6)	2.518 (3)	152 (5)
O1*W*—H1*A*···O72^{i}	0.90 (10)	2.11 (10)	3.008 (7)	179 (9)
O1*W*—H1*B*···O53^{ii}	0.93 (11)	2.13 (11)	3.055 (7)	180 (8)
N11—H11*A*···O53	0.81 (7)	2.29 (7)	3.018 (7)	151 (5)
N11—H11*A*···O2^{iii}	0.81 (7)	2.71 (7)	3.079 (7)	110 (6)
N11—H11*B*···O51^{iv}	0.90 (5)	2.19 (5)	3.089 (7)	179 (6)
N12—H12*A*···O72^{iii}	0.90 (4)	2.09 (4)	2.987 (7)	179 (6)
N12—H12*B*···O52^{v}	0.89 (5)	2.17 (5)	3.006 (6)	157 (4)
N21—H21*A*···O52^{vi}	0.83 (5)	2.52 (5)	3.127 (6)	131 (5)
N21—H21*A*···O2^{iii}	0.83 (5)	2.34 (5)	3.031 (6)	141 (5)
N21—H21*B*···O51^{vii}	0.89 (6)	2.21 (6)	3.003 (7)	149 (5)
N22—H22*A*···O71^{i}	0.90 (7)	1.94 (7)	2.801 (6)	159 (6)
N22—H22*B*···O52^{v}	1.00 (9)	2.22 (9)	3.043 (7)	139 (7)
N32—H32*A*···O1*W*	0.94 (9)	1.93 (9)	2.871 (8)	180 (8)
N32—H32*B*···O71^{iii}	0.81 (7)	2.06 (8)	2.826 (8)	157 (8)
C4—H4···O51	0.95	2.59	2.953 (5)	103

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*+1/2, −*y*, *z*+1/2; (iv) *x*−1/2, −*y*+1/2, −*z*+1; (v) *x*+1/2, −*y*+1/2, −*z*+1; (vi) *x*, *y*, *z*+1; (vii) *x*−1/2, −*y*+1/2, −*z*+2.

Figure 1

Fig. 1. Molecular configuration and atom naming scheme for (I). Atoms are shown as 30% probability ellipsoids

Figure 2

Fig. 2. Perspective view of the packing of (I) in the unit cell viewed down a, showing hydrogen-bonding associations as broken lines.